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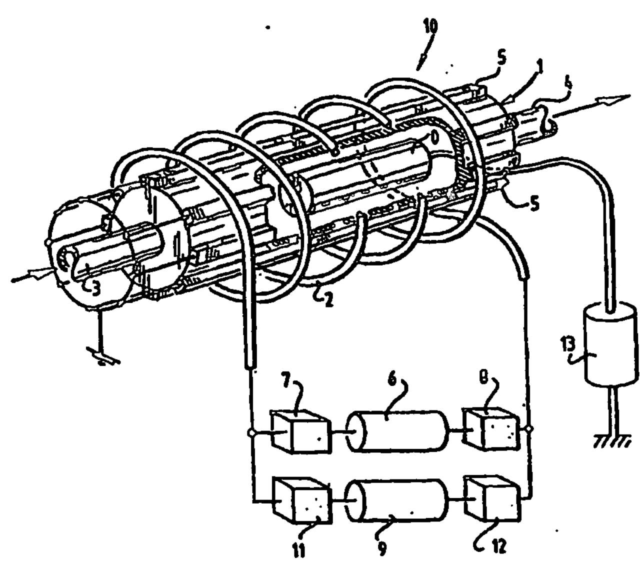

(57) Abstract

A method for processing a substrate of a certain material, wherein the material is inducted with heat by a relatively low frequency electrical current and wherein a relatively high frequency electrical current is used for further processing of said material. An apparatus for processing an object out of, or a certain amount of a certain material, wherein the device comprises: a reactor chamber (10) with an outer wall, which is at least partially made from non-conducting material; arranging means for arranging of the material in the reaction chamber; and an electric conductor (2), for generating an alternating electric field in the reactor, which is supplied next to the wall of the reactor.

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MULTI-FREQUENCY INDUCTIVE METHOD AND APPARATUS FOR THE PROCESSING OF MATERIAL

Chemical Vapour Deposition (CVD) is used on a large scale in industry for depositing layers of a certain material onto a substrate as general term, at typical temperatures of 800°C or more. By lower temperatures, Plasma Enhanced Chemical Vapour Deposition (PECVD) can be used. However this has the disadvantage that defects in uniformity can occur when depositing layers onto three dimensional objects. PECVD is thus mainly used for depositing layers onto flat surfaces. For applying thin films at low temperatures a so called Distributed Electronic Cyclotronic Resonnance Reactor (DECR) is also used for uniformity on 3-dimensions substrates. However DECR apparatus is complex and expensive for industrial applications, whilst at the same time the deposition rate is too low for most of the industrial applications.

The purpose of the present invention is to provide a method and apparatus wherein at relatively low temperatures, substantially under 800°C for the substrate, layers can be applied whilst achieving a high deposition rate, an excellent three dimensional uniformity and wherein the microstructure of the deposited layers can be accurately controlled and/or electrically conducting layers can be deposited.

Accordingly the present invention provides a method for the processing of a substrate of a certain material, wherein the material is heated by induction at relatively low frequency and wherein a relatively high frequency electrical current is used for further processing of said material.

In this context the word substrate comprises any object of any shape of a certain material and also a certain quantity of bulk material.

Further the present invention provides an apparatus for processing a substrate, wherein the apparatus comprises:

- a reactor chamber with an outer wall, which is at least partially made from non-electrically conducting material;
- arranging means for arranging of the substrate in the reaction chamber; and
- an electric conductor, for generating an alternating electric field in the reactor, which is provided adjacent to the wall of the reactor.

Further advantages, characteristics and details of the present invention will become clear with respects to the following description of a preferred embodiment thereof, and to the accompanying figure.

A preferred embodiment 10 of the apparatus according to the present invention for the carrying out of a preferred embodiment of the method according to the present invention, comprises a substantially cylindrical or tubelike reaction chamber 1, around which an electromagnetic winding 2 is arranged. An object O for instance made of metal, is suspended or otherwise arranged in the chamber. The object O can be made of conducting material. The wall 1 is built from electrically insulating material, for example alumina. Electrically conducting strips 5 are provided on or in the wall 1, the strips being preferably connected to ground. The electrically conducting strips 5 provide an uniform inductive electric field in the reaction chamber compensating for the limited length thereof. A reaction gas entry pipe 3, shown with an arrow, is disposed to a first side whilst on the opposite side a reaction gas exit pipe 4 is disposed.

A low frequency power generator 6 is provided between two filters 7 and 8 respectively in order to make possible inductive heating of the object O by the winding 2, low frequency here meaning a frequency smaller or equal to 10 kHz. Furthermore, a high frequency power generator 9 is arranged inbetween two filters 11 and 12 and connected to

the electromagnetic winding 2 for the generation of electromagnetic power, for example at a frequency of 13,56 MHz or more, which frequencies are used for plasma generation as is necessary for PECVD processing of the object O. The low frequency power generator 6 can, for example, supply a power of around 20 kWatts, whilst the high frequency power generator can supply, for instance, an operating power of around 5 kWatts. The object O is connected to a high frequency power bias generator 13, so that suitable operation for the accurate control of ion energy impinging on the surface of the object can occur. The bias generator 13 can be phase locked with the high frequency generator 9.

A first example of the method according to the present invention concerns the deposition of a cubic boron nitride film onto a conducting object. A gas mixture of N_2 with a flow of 90 sccm (standard cubic centimeters per minute), H_2 with a flow of 10 sccm and WF_6 with a flow of 5 sccm are supplied in the process chamber at a pressure of 800 Pascal. A plasma is inductively gererated with a radiofrequency adjustable power, at 13,56 MHz of roughly 100 Watt which is supplied by the generator 9, whereby a power density of 500 mW/cm^2 is realized near the object by the power generator 13. The power generator 13 is required to control the bias voltage to the object for instance to a few hundreds of volts. The object O can be kept accurately within a temperature range of 200-500°C with the aid of the low frequency power generator. A temperature sensor, not shown, is disposed within the reaction chamber and is coupled to a control, not shown, which controls power from the low frequency generator 6.

A second preferred embodiment of the method according to the present invention concerns the deposition of a diamond like coating onto an electrically conducting object with a total gas flow of 80 sccm, wherein the gas flow is substantially H_2 comprising 7 mol.% CO and 2,2 mol.% O_2 at a pressure of 250 Pascal. The high frequency generator 9 generates the plasma at an adjustable power level of a few

hundreds Watts. The power generator 13 will be used to adjust the bias of the object O. With this process, the temperature is preferably kept in the range of 400-750°C by accurate inductive heating ensured by the low-frequency power generator 6. In this second embodiment a reactor using inductive coupling is provided wherein the voltages across the plasma are low and therefore the ions have low energy.

The low frequency induction is not, or at any rate only in a small amount, influenced by the plasma present. The generated high frequency energy is virtually completely absorbed by the resulting plasma, and does not directly interact with the substrate.

The above described embodiments of the present invention provide a great number of advantages of which an important number, are the following:

- the plasma density can be very high, for example up to 10^{12} per cm^2;
- the pressure range can be very wide, for instance from 10^{-4} to 1 Torr;
- a high level of three dimensional homogenity is achieved in the plasma;
- the substrate, can be provided with an independant bias voltage which makes possible the control of the microstructure of the deposited layer;
- by adjusting the frequencies, an easy scale-up or scale-down of the process chamber can be achieved;
- reactor wall sputtering is prevented due to the very low ion energies which therefore ensures a clean technology;
- the heating efficiency can be large due to the fact that not much energy loss occurs during heating;
- the heating can occur quickly due to the fact that the object is directly heated by so called eddy currents without entailing a large amount of thermal inertia;
- the temperature regulation of the object or the substrate, can take place easily and accurately;

- the fact that the wall remains cold, prevents wall deposition and avoid thus electrical screening of the applied inductive fields allowing metal deposition processes;

- the apparatus can be kept compact, whilst at the same time many bias parameters, such as differing frequencies and temperatures, are possible; the production costs are expected to be low, the maintenance easy, and the operation is expected to be exceptional due to the simplicity and low break-down susceptibility.

The requested rights are in no way limited by the hereabove described preferred embodiments of the invention, but are rather defined by the following claims. In that respect it will be especially noted by one skilled in the art that an intended non limiting variation with respects to the embodiments is concerned with allowing transfer of low frequency and high frequency electromagnetic power respectively, by means of separate coil windings, as well as other processes than PECVD being possible with the apparatus and method according to the present invention.

There are for example lots of promising applications in the field of metallurgy, where the multi-frequency induction can be used to achieve process performance out of reach by any other means.

CLAIMS

1. A method for processing a substrate of a certain material, wherein the material is inducted with heat by a relatively low frequency electrical current and wherein a relatively high frequency electrical current is used for further processing of said material.

2. A method according to claim 1, wherein the relatively high frequency is capable of generating plasma from a supplied gas and wherein the processing is PECVD.

3. A method according to claims 1 or 2, wherein the object is an electrically conducting object that is for instance made of metal.

4. A method according to claims 1, 2 or 3, wherein a gas mixture of N_2, H_2 and WF_6 is supplied into the reaction chamber.

5. An apparatus according to claims 1, 2 or 3, wherein a gas mixture comprising H_2, CO and O_2 is supplied to the reactor.

6. An apparatus for processing an object out of, or a certain amount of a certain material, wherein the device comprises:

- a reactor chamber with an outer wall, which is at least partially made from non-conducting material;
- arranging means for arranging of the material in the reaction chamber; and
- an electric conductor, for generating an alternating electric field in the reactor, which is supplied next to the wall of the reactor.

7. An apparatus according to claim 6, wherein the reaction chamber is provided with one or more gas entries and one or more gas exits.

8. An apparatus according to claim 6 or 7, wherein strips of electrically conductive material are provided in, near or on the outer wall.

9. An apparatus according to any of the claims 6, 7 or 8, provided with a generator for building up a bias voltage to the object to be arranged in the reaction chamber.

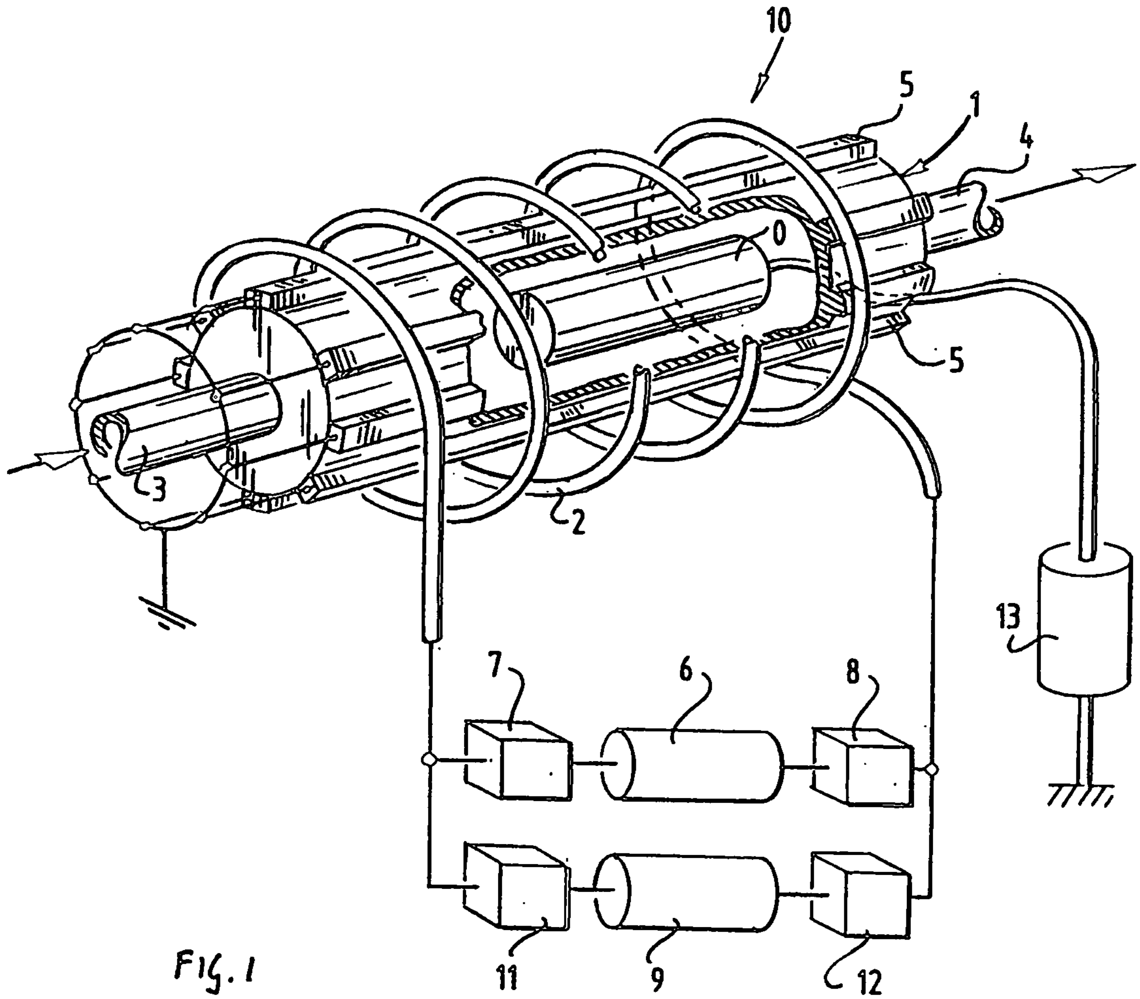

FIG. 1

INTERNATIONAL SEARCH REPORT

Inter nal Application No
PCT/EP 95/01522

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C23C16/50 C23C16/46 H01J37/32

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C23C H01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US-A-4 388 344 (A.J. SHUSKUS) 14 June 1983 see figure 3 ---	1,2,6,7
X	DATABASE WPI Section Ch, Week 8738 Derwent Publications Ltd., London, GB; Class G08, AN 87-268976 & JP-A-62 188 783 (SANYO ELECTRIC KK) , 18 August 1987	1-3
Y	see abstract --- -/--	4,6,7,9

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
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Name and mailing address of the ISA	Authorized officer
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JAPANESE JOURNAL OF APPLIED PHYSICS, JULY 1969, JAPAN, VOL. 8, NR. 7, PAGE(S) 876 - 882, ISSN 0021-4922, KUWANO Y 'Some properties of silicon nitride films produced by radio frequency glow discharge reaction of silane and nitrogen' see figure 1	6,7
Y	US-A-4 664 747 (A. SEKIGUCHI) 12 May 1987 see column 3, line 10 - line 44	6,7,9
A	see column 6, line 63 - column 7, line 27	4
A	EP-A-0 457 076 (HITACHI LTD) 21 November 1991 see example 1	5
A	US-A-2 793 140 (B. OSTROFSKY) 21 May 1957 see column 4, line 1 - line 9	8
A	THIN SOLID FILMS, 15 APRIL 1980, SWITZERLAND, VOL. 67, NR. 2, PAGE(S) 321 - 324, ISSN 0040-6090, OLCAYTUG F ET AL 'A low temperature process for the reactive formation of Si/sub 3/N/sub 4/ layers on InSb' see figure 1	9
A	PATENT ABSTRACTS OF JAPAN vol. 005 no. 086 (E-060) ,5 June 1981 & JP,A,56 033839 (HITACHI LTD) 4 April 1981, see abstract	1-9
Y	PATENT ABSTRACTS OF JAPAN vol. 013 no. 169 (C-587) ,21 April 1989 & JP,A,63 317676 (SHARP CORP) 26 December 1988, see abstract	4

INTERNATIONAL SEARCH REPORT

Information on patent family members

Interna l Application No
PCT/EP 95/01522

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
US-A-4388344	14-06-83	NONE		
US-A-4664747	12-05-87	JP-C-	1786581	10-09-93
		JP-B-	4071575	16-11-92
		JP-A-	61222534	03-10-86
EP-A-0457076	21-11-91	JP-A-	4214094	05-08-92
US-A-2793140	21-05-57	NONE		